# EIGHTEENTH QUARTERLY PROGRESS REPORT

6-1-64 - 9-30-64

on

NA SW-101

INVESTIGATION OF MECHANICAL PROPERTIES OF CHROMIUM, CHROMIUM-RHENIUM, AND DERIVED ALLOYS

to

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

October 1, 1964

by

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## ABSTRACT

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Optical and electron replica factographic observations were made on specimens of tungsten, W-3Re and W-5Re which had been factured by bending at temperatures varying from 0 F to 700 F. It was found that for recrystallized material, additions of rhenium increased the percentage of cleavage failure occuring but had no noticeable effect on the morphology of grain-boundary precipitates. The major effect of rhenium is thought to be on the recrystallized grain size and the stress at which fracture is initiated. For wrought material, resistance to cleavage failure arises from delamination of the wrought structure. The implications of these observations are discussed.

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# INVESTIGATION OF MECHANICAL PROPERTIES OF CHROMIUM, CHROMIUM-RHENIUM, AND DERIVED ALLOYS

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#### INTRODUCTION

It has been found that additions of rhenium to chromium, molybdenum, and tungsten can greatly enhance the ductility of commercially pure base materials. The effect is most pronounced at fairly high alloying concentrations, for example, in Cr-35 at. % Re, Mo-33 at. % Re, and W-23 at. % Re. In view of the high cost of rhenium, however, the enhancement of ductility by large alloying additions cannot be considered a practical solution to the brittleness of these refractory metals. Work has, therefore, been done on dilute alloys of chromium(1)\* and tungsten(2,3) with rhenium, in order to determine whether the beneficial effect of rhenium persists to lower alloying levels. In the case of chromium, it was found that dilute additions of rhenium produced only a normal solid-solution embrittlement. Dilute W-Re alloys, however, have shown<sup>(3)</sup> improved ductility over that of unalloyed tungsten. To try to get some idea of the mechanism of this improvement due to rhenium additions, a fractographic examination was made of specimens of tungsten and dilute W-Re alloy specimens. The objective of the work was to determine how rhenium modifies the fracture mode in tungsten and also, if possible, to determine how rhenium modifies the precipitate morphology.

#### EXPERIMENTAL PROCEDURE

Samples of tungsten, W-3Re, and W-5Re in various heat-treated conditions were available from earlier bend-test experiments performed at the NASA Lewis Research Laboratory. The samples had been tested at temperatures in the range 0 to 700 F in determination of ductile-brittle transition temperatures. To examine fracture surfaces produced at a common test temperature, one recrystallized sample of each composition was fractured again at room temperature. The broken specimens were subjected to optical fractographic and metallographic examinations and in addition to electron replica fractography. For the latter operation, replicas were made by pressing cellulose acetate sheet softened with acetone onto the fracture surface, where it was allowed to harden for several minutes. The replica was then stripped from the fracture surface by hand and placed in a vacuum evaporator, where Pt-C shadowing mixture was deposited at 45 degrees to the replica surface. Carbon was then deposited normal to the replica. Finally, the plastic backing was dissolved in 3:1 ethanol:acetone mixture and the carbon replica washed in pure acetone. The replicas were mounted on a 200-mesh grid and examined in an RCA electron microscope.

<sup>\*</sup>References are listed at end of report.

The experimental observations were designed to provide information on the following points:

- (1) Fracture mode in:
  - (a) Tungsten (recrystallized)
  - (b) W-3Re (wrought and recrystallized)
  - (c) W-5Re (wrought and recrystallized).

From these observations, the effect of rhenium on the susceptibility of tungsten to cleavage fracture should become clear.

- (2) Precipitate morphology in:
  - (a) Tungsten (recrystallized)
  - (b) W-3Re (recrystallized)
  - (c) W-5Re (recrystallized).

From these observations, it should be possible to learn whether rhenium exerts a noticeable effect on the nature of grain-boundary precipitates in tungsten.

#### EXPERIMENTAL OBSERVATIONS

Table 1 lists the heat treatments, test temperatures, and fracture modes of 26 specimens of tungsten, W-3Re, and W-5Re which were examined fractographically. For purposes of comparison the experimental observations on the three recrystallized materials will be reported together, followed by observations on the two wrought alloys.

#### Recrystallized Materials

Figure 1 shows the grain structures of tungsten annealed for 1 hour at 3500 F and W-3Re and W-5Re annealed for 1 hour at 3600 F. It is apparent that rhenium additions significantly retard recrystallization, since although annealed at a lower temperature, the grain size of the unalloyed tungsten is much larger than for the alloys. The general fracture characteristics of the three materials are summarized in the following paragraphs.

## Tungsten

The tungsten specimens tested after 1-hour vacuum anneals at 3500 F, 3800 F, 4000 F, and 4200 F, had grain structures containing approximately 200, 60, 25, and 14 grains in the specimen cross section. In accordance with previous experience of tension tests in chromium, it was found that the transition temperature decreased as the grain size increased.

TABLE 1. SUMMARY OF BEND-TEST SPECIMENS EXAMINED FRACTOGRAPHICALLY

Specimen         Composition         Heat Treatment, Annealing Time         Test of Temperature and Temperature and Specimen and Temperature					Sp	Specimen Appearance	
Composition         Annealing Time         Temperature         of         Fracture Mode(a)           Tungsten         1 hr 3500 F         < 750         Slightly         95% GB, 5% CI           Tungsten         1 hr 3500 F         < 750         Bent         90% GB, 10% CI           Tungsten         1 hr 3800 F         < 750         Bent         90% GB, 10% CI           Tungsten         1 hr 3800 F         < 7705         Bent         4 CJ,           Tungsten         1 hr 4000 F         < 695         Slightly         1 CJ, acets           Tungsten         1 hr 4000 F         < 695         Slightly         1 CJ, acets           Tungsten         1 hr 4000 F         < 695         Slightly         1 CJ, acets           Tungsten         1 hr 4200 F         < 695         Slightly         1 CJ, acets           Tungsten         1 hr 4200 F         < 630         None         100% GB           Ww-3 Re         1 hr 4200 F         < 630         None         Dolan and Cl           Ww-3 Re         1 hr 1800 F         < 630         None         Dolan and Cl           Ww-3 Re         1 hr 3600 F         < 630         None         Dolan and Cl           Ww-3 Re         1 hr 3600 F         < 630			Heat Treatment,	Test	Degree		Transition
Tungsten         1 hr 3500 F         Vintested <th>Specimen</th> <th>Composition</th> <th>ealing Ti Tempera</th> <th>Temperature, F</th> <th>of Bending</th> <th>Fracture Mode(a)</th> <th>Temperature, F</th>	Specimen	Composition	ealing Ti Tempera	Temperature, F	of Bending	Fracture Mode(a)	Temperature, F
Tungsten Ihr 3500 F < 750 Slightly 95% GB, 5% CI Tungsten Ihr 3500 F < 750 Bent 90% GB, 10% CI Tungsten Ihr 3800 F < 705 None 50% pre-existing crack, 26 GB, 13 CI facets  Tungsten Ihr 4000 F 695 Slightly 1 CI, 28 GB facets,  Tungsten Ihr 4000 F 695 Slightly 1 CI, 4 GB facets  Tungsten Ihr 4000 F 695 Slightly 1 CI, 28 GB facets  Tungsten Ihr 4200 F 630 Slightly 100% GB Tungsten Ihr 4200 F 630 Slightly 15 CI facets  Tungsten Ihr 4200 F 630 Slightly 15 CI facets  Tungsten Ihr 4200 F 630 Slightly 15 CI facets  Tungsten Ihr 4200 F 630 Slightly 15 CI facets  W-3 Re Ihr 2600 F 630 Slightly 15 CI facets  W-3 Re Ihr 2600 F 70 None Ditto  W-3 Re Ihr 2600 F 70 None Ditto  W-3 Re Ihr 3600 F 70 None CO% CI Some delam  W-5 Re Ihr 3600 F 425 None CO% CI Delam and CI  W-5 Re Ihr 3600 F 425 None CO% CI Delam and CI  W-5 Re Ihr 3600 F 425 None CO% CI Delam and CI  W-5 Re Ihr 3600 F 630 None CO% CI OP CI  W-5 Re Ihr 3600 F 630 None CO% CI OP CI  W-5 Re Ihr 3600 F 630 None CO% CI OP CI  W-5 Re Ihr 3600 F 630 None CO% CI OP CI  W-5 Re Ihr 3600 F 630 None CO% Ditto  W-5 Re Ihr 3600 F 630 None CO% CI OP None CO% Ditto  W-5 Re Ihr 3600 F 630 None CO% CI OP	-	Tungeton	3500	Untested	;	l t	
Tungsten I hr 3800 F < 750 Bent 90% GB, 10% CI Tungsten I hr 3800 F < 705 Bent 26 GB, 13 CI facets  Tungsten I hr 4000 F < 695 Slightly 1 CI, 41 GB facets  Tungsten I hr 4000 F < 695 Slightly 100% GB  Tungsten I hr 4000 F < 695 Slightly 100% GB  Tungsten I hr 4200 F < 630 None 100% GB  Tungsten I hr 4200 F < 630 Slightly 100% GB  Tungsten I hr 4200 F < 630 Slightly 100% GB  Tungsten I hr 4200 F < 630 Slightly 100% GB  Tungsten I hr 4200 F < 630 Slightly 100% GB  Tungsten I hr 4200 F < 630 Slightly 100% GB  Tungsten I hr 4200 F < 630 Slightly 100% GB  W-3 Re I hr 4200 F < 630 None Ditto  W-3 Re I hr 1800 F < 630 None Gelam Ditto  W-3 Re I hr 2600 F 150 None 60% GB, 40% CI  W-3 Re I hr 3600 F 150 None 60% GB, 50% CI  W-5 Re As rolled 53 Bent Ditto  W-5 Re As rolled 53 Bent Ditto  W-5 Re I hr 3600 F 600 Slightly I 1000 Sl	, ,	Tungsten	hr 3500	< 750	Slightly	95% GB, 5% Cl	750
Tungsten I hr 3800 F < 705 None 50% pre-existing crack, Tungsten I hr 3800 F > 705 Bent 4 G, Tungsten I hr 4000 F 695 Slightly 1 G, Tungsten I hr 4000 F < 695 Slightly 1 G, Tungsten I hr 4200 F < 630 Slightly 1 G, Tungsten I hr 4200 F < 630 Slightly 1 G, Tungsten I hr 4200 F < 630 Slightly 1 G, Tungsten I hr 4200 F < 630 None 1 G, Tungsten I hr 4200 F < 630 None 1 G, W-3 Re I hr 1800 F < 630 None Dinto Dinto W-3 Re I hr 2600 F 70 None 1 Largely CI, Some delam W-3 Re I hr 2600 F 70 None 60% GB, 50% CI None W-3 Re I hr 3600 F 425 None 60% GB, 55% CI None CM, 56% CI None	7 %	Tungsten	hr 3500	> 750	Bent	90% GB, 10% CI	750
Tungsten         1 hr 3800 F         > 705         Bent         4 GB facets,           Tungsten         1 hr 4000 F         695         Slightly         1 CJ,           Tungsten         1 hr 4000 F         > 695         Slightly         24 GB, 8 CI           Tungsten         1 hr 4200 F         > 695         Slightly         100% GB           Tungsten         1 hr 4200 F         > 695         Slightly         100% GB           Tungsten         1 hr 4200 F         > 695         Slightly         100% GB           Tungsten         1 hr 4200 F         > 695         Slightly         100% GB           Tungsten         1 hr 4200 F         > 630         Slightly         100% GB           W-3 Re         1 hr 4200 F         > 630         None         Ditto           W-3 Re         1 hr 1800 F         0         Bent         Ditto           W-3 Re         1 hr 2600 F         70         None         60% GB, 40% CI           W-3 Re         1 hr 3600 F         405         None         45% GB, 55% CI           W-5 Re         1 hr 3600 F         405         None         50% GB, 50% CI           W-5 Re         1 hr 3600 F         0         Bent         Ditto <tr< td=""><td>7 4</td><td>Tungsten</td><td>hr 3800</td><td>&lt; 705</td><td>None</td><td>50% pre-existing crack, 26 GB, 13 Cl facets</td><td>705</td></tr<>	7 4	Tungsten	hr 3800	< 705	None	50% pre-existing crack, 26 GB, 13 Cl facets	705
Tungsten         1 hr 4000 F         695         Slightly         1 CJ, 41 GB facets           Tungsten         1 hr 4000 F         > 695         Slightly         24 GB, 8 Cl           Tungsten         1 hr 4200 F         < 630	7.	Tungsten	hr 3800		Bent	4 Cl, 28 GB facets,	705
Tungsten         1 hr 4000 F         695         Slightly         24 GB, 8 CI           Tungsten         1 hr 4000 F         > 695         Slightly         100% GB           Tungsten         1 hr 4200 F         < 630	9	Tungsten	hr 4000	969	Slightly	1 Cl, 41 GB facets	969
Tungsten         1 hr 4000 F         695         Slightly         24 GB, 6 GB           Tungsten         1 hr 4000 F         < 695				1		,	907
Tungsten         1 hr 4000 F         > 695         Slightly         100% GB           Tungsten         1 hr 4200 F         < 630	2	Tungsten	4000	645	Slightly	24 GB, 8 CI	660
Tungsten         1 hr 4200 F         < 630         None         100% GB           Tungsten         1 hr 4200 F         630         Slightly         15 GB, 1 Cl facet           Tungsten         1 hr 4200 F         < 630	8	Tungsten	4000		Slightly	100% GB	669
Tungsten         1 hr 4200 F         630         Slightly         15 GB, 1 Cl facet           Tungsten         1 hr 4200 F         > 630         Bent         12 GB, 2 Cl facets           Tungsten         1 hr 4200 F         < 630	6	Tungsten	4200		None	$100\%~\mathrm{GB}$	630
Tungsten         1 hr 4200 F         > 630         Bent         12 GB, 2 Cl facets           Tungsten         1 hr 4200 F         < 630	10	Tungsten	4200	630	Slightly	15 GB, 1 Cl facet	630
Tungsten         1 hr 4200 F         < 630         None         pre-existing crack           W-3 Re         1 hr 1800 F         25         None         Ditto           W-3 Re         1 hr 2600 F         70         None         Largely Cl, Some delam           W-3 Re         1 hr 2600 F         150         None         Ditto           W-3 Re         1 hr 3600 F         300         None         60% GB, 40% Cl           W-3 Re         1 hr 3600 F         400         None         50% GB, 50% Cl           W-3 Re         1 hr 3600 F         425         None         45% GB, 55% Cl           W-5 Re         1 hr 3600 F         25         Bent         Delam and Cl           W-5 Re         1 hr 2000 F         0         Slightly         "           W-5 Re         1 hr 2000 F         0         Slightly         "           W-5 Re         1 hr 3600 F         400         None         60% Cl, 40% GB           W-5 Re         1 hr 3600 F         300         None         60% Ditto           W-5 Re         1 hr 3600 F         400         None         60% Ditto           W-5 Re         1 hr 3600 F         400         None         60% Ditto	11	Tungsten	4200		Bent	12 GB, 2 Cl facets	630
W-3 Re         1 hr 1800 F         0         Bent         Ditto           W-3 Re         1 hr 2600 F         70         None         Largely Cl, Some delam           W-3 Re         1 hr 2600 F         70         None         Ditto           W-3 Re         1 hr 3600 F         300         None         60% GB, 40% Cl           W-3 Re         1 hr 3600 F         400         None         50% GB, 50% Cl           W-5 Re         1 hr 3600 F         425         None         45% GB, 55% Cl           W-5 Re         As rolled         25         Bent         Delam and Cl           W-5 Re         1 hr 2000 F         0         Slightly         "           W-5 Re         1 hr 2000 F         0         Slightly         "           W-5 Re         1 hr 3600 F         400         None         60% Cl, 40% GB           W-5 Re         1 hr 3600 F         400         None         60% Ditto           W-5 Re         1 hr 3600 F         400         None         60% Ditto           W-5 Re         1 hr 3600 F         400         None         60% Ditto	12	Tungsten	4200		None	pre-existing crack	630
W-3 Re       None       Ditto         W-3 Re       1 hr 2600 F       70       None       Largely Cl, Some delam         W-3 Re       1 hr 2600 F       150       None       Ditto         W-3 Re       1 hr 3600 F       300       None       60% GB, 40% Cl         W-3 Re       1 hr 3600 F       425       None       45% GB, 55% Cl         W-5 Re       As rolled       25       Bent       Ditto         W-5 Re       As rolled       53       Bent       Ditto         W-5 Re       1 hr 2000 F       0       Slightly       "         W-5 Re       1 hr 3600 F       300       None       60% Cl, 40% GB         W-5 Re       1 hr 3600 F       300       None       60% Ditto         W-5 Re       1 hr 3600 F       400       None       60% Ditto         W-5 Re       1 hr 3600 F       400       None       60% Ditto	13	W-3 Re	1800	0	Bent	Delam and Cl	7.5
W-3 Re       1 hr 2600 F       70       None       Largely Cl, Some delam         W-3 Re       1 hr 2600 F       150       None       Ditto         W-3 Re       1 hr 3600 F       300       None       60% GB, 40% Cl         W-3 Re       1 hr 3600 F       425       None       45% GB, 55% Cl         W-5 Re       1 hr 3600 F       25       Bent       Ditto         W-5 Re       1 hr 2000 F       0       Bent       Ditto         W-5 Re       1 hr 2000 F       0       Slightly       "         W-5 Re       1 hr 3600 F       400       None       60% Cl, 40% GB         W-5 Re       1 hr 3600 F       400       None       60% Cl, 40% GB         W-5 Re       1 hr 3600 F       400       None       60% Ditto         W-5 Re       1 hr 3600 F       450       None       60% Ditto	14			2.5	None	Ditto	7.5
W-3 Re       1 hr 2600 F       70       None       Largely Cl, Some delam         W-3 Re       1 hr 3600 F       300       None       60% GB, 40% Cl         W-3 Re       1 hr 3600 F       400       None       50% GB, 40% Cl         W-3 Re       1 hr 3600 F       425       None       45% GB, 55% Cl         W-5 Re       As rolled       25       Bent       Delam and Cl         W-5 Re       1 hr 2000 F       0       Bent       1         W-5 Re       1 hr 2000 F       0       Slightly       "         W-5 Re       1 hr 3600 F       300       None       60% Cl, 40% GB         W-5 Re       1 hr 3600 F       400       None       60% Ditto         W-5 Re       1 hr 3600 F       450       None       60% Ditto	15			50	Bent	Ξ	75
W-3 Re       1 hr 2600 F       150       None       Ditto         W-3 Re       1 hr 3600 F       300       None       60% GB, 40% CI         W-3 Re       1 hr 3600 F       425       None       45% GB, 55% CI         W-5 Re       As rolled       25       Bent       Delam and Cl         W-5 Re       As rolled       53       Bent       Ditto         W-5 Re       1 hr 2000 F       0       Slightly       "         W-5 Re       1 hr 3600 F       300       None       60% Cl, 40% GB         W-5 Re       1 hr 3600 F       400       None       60% Ditto         W-5 Re       1 hr 3600 F       450       None       60% Ditto	16		2600	7.0	None	Largely Cl, Some delam	200
W-3 Re       1 hr 3600 F       300       None       60% GB, 40% CI         W-3 Re       1 hr 3600 F       425       None       50% GB, 50% CI         W-5 Re       As rolled       25       Bent       Delam and CI         W-5 Re       As rolled       53       Bent       Ditto         W-5 Re       1 hr 2000 F       0       Bent       "         W-5 Re       1 hr 2000 F       0       Slightly       "         W-5 Re       1 hr 3600 F       300       None       60% CI, 40% GB         W-5 Re       1 hr 3600 F       400       None       60% Ditto         W-5 Re       1 hr 3600 F       450       None       60% Ditto	1.7	-3	2600	150	None	Ditto	200
W-3 Re       1 hr 3600 F       400       None       50% GB, 50% CI         W-3 Re       As rolled       25       Bent       Delam and CI         W-5 Re       As rolled       53       Bent       Ditto         W-5 Re       1 hr 2000 F       0       Bent       "         W-5 Re       1 hr 2000 F       0       Slightly       "         W-5 Re       1 hr 3600 F       300       None       60% CI, 40% GB         W-5 Re       1 hr 3600 F       400       None       60% Ditto         W-5 Re       1 hr 3600 F       450       None       60% Ditto	18		3600	300	None	60% GB, 40% Cl	450
W-3 Re       1 hr 3600 F       425       None       45% GB, 55% Cl         W-5 Re       As rolled       25       Bent       Ditto         W-5 Re       1 hr 2000 F       0       Bent       "         W-5 Re       1 hr 2000 F       0       Slightly       "         W-5 Re       1 hr 3600 F       300       None       60% Cl, 40% GB         W-5 Re       1 hr 3600 F       400       None       60% Ditto         W-5 Re       1 hr 3600 F       450       None       60% Ditto	19		3600	400	None	50% GB, 50% Cl	450
W-5 Re       As rolled       25       Bent       Delam and Cl         W-5 Re       1 hr 2000 F       0       Bent       "         W-5 Re       1 hr 2000 F       0       Slightly       "         W-5 Re       1 hr 3600 F       300       None       60% Cl, 40% GB         W-5 Re       1 hr 3600 F       400       None       60% Ditto         W-5 Re       1 hr 3600 F       450       None       60% Ditto	20		3600	425	None	45% GB, 55% Cl	450
W-5 Re       As rolled       53       Bent       Ditto         W-5 Re       1 hr 2000 F       0       Slightly       "         W-5 Re       1 hr 3600 F       300       None       60% Cl, 40% GB         W-5 Re       1 hr 3600 F       400       None       60% Ditto         W-5 Re       1 hr 3600 F       450       None       "	2.1			2.5	Bent	Delam and Cl	-25
W-5 Re       1 hr 2000 F       0       Bent       "         W-5 Re       1 hr 3600 F       300       None       60% Cl, 40% GB         W-5 Re       1 hr 3600 F       450       None       60% Ditto         W-5 Re       1 hr 3600 F       450       None       "	22	5		53	Bent	Ditto	2
W-5 Re       1 hr 2000 F       0       Slightly       "         W-5 Re       1 hr 3600 F       300       None       60% Cl, 40% GB         W-5 Re       1 hr 3600 F       450       None       60% Ditto         W-5 Re       1 hr 3600 F       450       None       "	23	5	2000	0	Bent	F	0
W-5 Re       1 hr 3600 F       300       None       60% Cl, 40% GB         W-5 Re       1 hr 3600 F       450       None       60% Ditto         W-5 Re       1 hr 3600 F       450       None       "	24	-5 R	2000	0	Slightly	Ξ	0
W-5 Re 1 hr 3600 F 400 None 60% Ditto W-5 Re 1 hr 3600 F 450 None "	2.5	-5 R	3600	300	None	60% C1, 40% GB	200
W-5 Re 1 hr 3600 F 450 None "	26	-5 R	3600	400	None	60% Ditto	200
	2.7	-5 R	3600	450	None	Ξ	200

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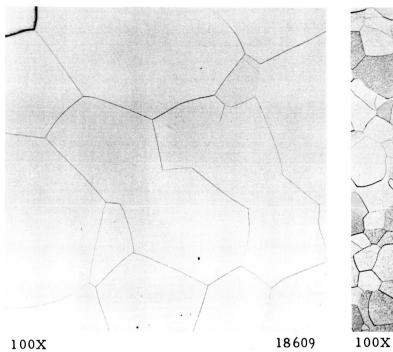
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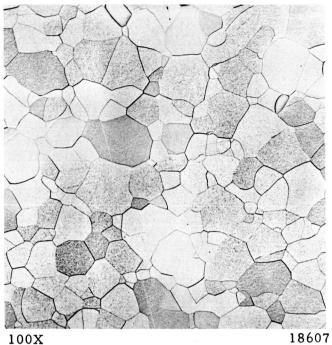
TABLE 1. (Continued)

	Transition	remperature, F		1	:	1	
Specimen Appearance		Fracture Mode(a)		90% GB, 10% Cl	30% GB, 30% Cl	20% GB, 80% CI	
Specir	Degree	ot Bending		None	None	None	
	Test	Temperature, F	•	7.0	70	7.0	
	Heat Treatment,	Annealing Time	and remperature	1 hr 3500 F	1 hr 3600 F	1 hr 3600 F	
			Composition	Tungsten	W-3 De	W-5 Re	
			Specimen	7	0 0	26	

(a) Fracture mode is described for most specimens as the percentage of grain-boundary (GB) and cleavage (Cl) fracture or the number of grain-boundary and cleavage facets, Delam = delaminations.

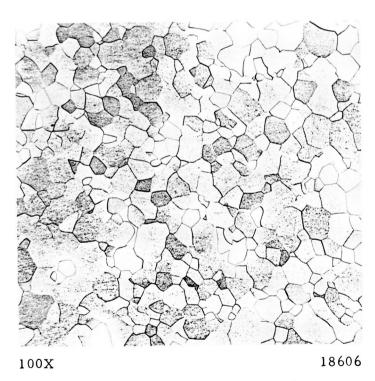
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a. Tungsten, Annealed 1 Hour at 3500 F.

b. W-3Re, Annealed 1 Hour at 3600 F



c. W-5Re, Annealed 1 Hour at 3600 F.

FIGURE 1. STRUCTURES OF RECRYSTALLIZED TUNGSTEN, W-3Re, AND W-5Re

Examination of the fracture surfaces by optical microscopy showed that the fracture surfaces were largely grain boundary and that the relative proportion of grain-boundary failure to cleavage failure did not vary much with grain size. There was some slight evidence that if grain size did affect the fracture mode, then coarse-grained material tended to fail completely by grain-boundary fracture, whereas the finer grained material tended to exhibit approximately 90% grain-boundary: 10% cleavage failure. An evaluation of the effect of grain size was obscured by the effect of prestrain on the failure mode, since there was a tendency for specimens which had been bent prior to fracture to exhibit more cleavage failure than those which were not bent. This can be seen by comparison of Specimens 2 and 3, and 9 and 11 of Table 1\*. This suggests that there is perhaps more plastic strain associated with slip-induced cleavage failures than with grain-boundary parting.

For all grain sizes, it was generally found that whenever grain-boundary and cleavage facets occurred on the same fracture surface there was a strong tendency for the larger grains to have cleaved and for the smaller ones to have parted at the boundary. Furthermore, a very common feature of the cleavage facets was the appearance of chevron markings similar to those shown in Figure 2a. Laue back-reflection, X-ray photographs on such facets showed the crystallographic relationships included in Figure 2a. These chevron markings are the same as those found in single-crystal molybdenum, chromium, and tungsten and are thought to represent a mechanism of slip-induced fracture. (4)\*\*

One reproducible feature of the cleavage facets was the change in nature of the river lines which occurred as cleavage failure propagated from the tensile side of the specimen to the compression side. An example of a cleavage facet which spans both tension and compression sides is shown in Figure 2b, a higher magnification photograph of the facet shown in Figure 2a. The sharp cleavage lines on the tension side of the specimen turn through approximately 90° as the original neutral axis is crossed, and they become much more wavy and coarse.

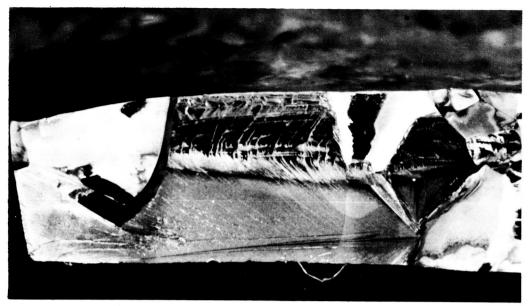
The preferential sites for the initiation of cleavage were found to be at the intersections of grain-boundary faces, as shown schematically in Figure 3. Both cleavage facets shown in Figure 3 had fracture origins located at the intersection of a grain corner with the cleavage plane. In plan, the point of initiation would be a triple point.

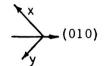
The precipitate morphology was approximately the same for all the recrystallized samples, regardless of annealing treatment. Second-phase particles were found preferentially on grain boundaries, and under the optical microscope were found to be roughly spherical. Figure 4 shows an exposed grain broundary at 250X and 750X. In no case was there any evidence under the optical microscope for a grain-boundary film of precipitate. However, there were examples of grain boundaries which showed colored interference effects next to grain boundaries which appeared white under reflected light. It is believed that the colored grain boundaries are covered with a contaminating film formed after testing and that the orientation determines whether or not a particular grain boundary is suitable for preferential oxidation or corrosion. At 1000X under the optical microscope this film appeared to be generally continuous but with a rough surface. This effect makes interpretation of electron replica fractographs extremely difficult because of the need to distinguish between oxide films formed by precipitation at the internal grain boundaries and those formed on exposed grain

<sup>\*</sup>No valid conclusions can be drawn for Specimens 4 and 12 because of the perturbing influence on the fracture mode of large pre-existing cracks.

See Figure 1 on page 2 of the Seventeenth Quarterly Progress Report to NASA on this contract.

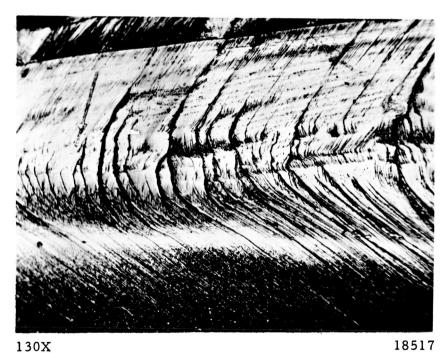
BATTELLE MEMORIAL INSTITUTE





40X 18396

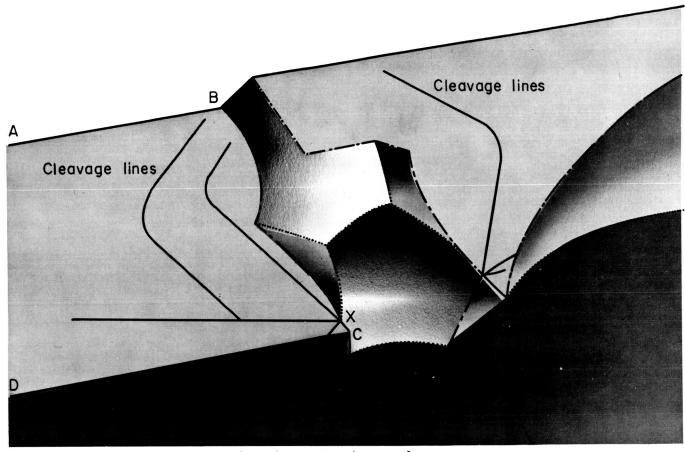
a. Central Line of Chevron is Along (010); Directions x and y Pass Through  $(\overline{112})$  and  $(\overline{112})$ , Respectively.



b Enlargement of Facet Above Showing Change in

b. Enlargement of Facet Above Showing Change in Cleavage Lines Across the Neutral Axis.

FIGURE 2. (001) CLEAVAGE FACET ON SPECIMEN 5,, SHOWING CHEVRON



Specimen tension surface

Grain boundary/grain boundary or grain boundary/surface join

Cleavage/cleavage or cleavage/surface join

Grain boundary/cleavage join

FIGURE 3. SCHEMATIC DRAWING OF CLEAVAGE-FAILURE ORIGIN IN SPECIMEN 7

Chevron heads at X and Y.

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9



250X 18511

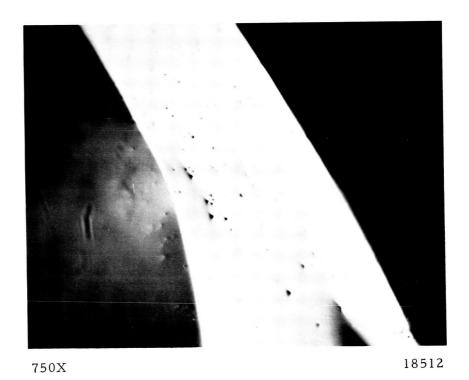


FIGURE 4. GLOBULAR PRECIPITATES AT EXPOSED GRAIN BOUNDARY ON FRACTURE SURFACE OF SPECIMEN 2 AT TWO MAGNIFICATIONS

boundaries after fracture. Examination of the surface of Specimen 3 immediately after fracture at room temperature showed evidence of only globular precipitates on the grain boundaries. Thus, the only grain-boundary precipitates which it is felt are typical of those existing prior to fracture are the globular type. An electron replica photograph of such a precipitate is shown in Figure 5.

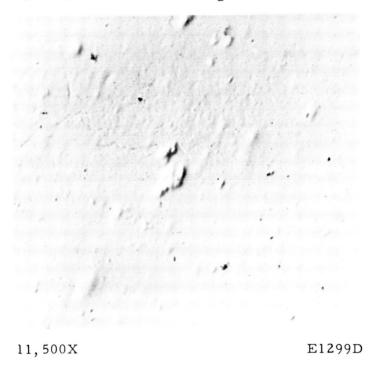


FIGURE 5. GLOBULAR PRECIPITATES ON TUNGSTEN GRAIN BOUNDARIES EXPOSED BY FRACTURE OF SPECIMEN 3 AT ROOM TEMPERATURE

On examining replicas of Specimen 5, taken from the general area shown in the upper half of Figure 2, it was found that occasionally precipitates had been extracted with the replica. A montage showing some extracted precipitates and some impressions on the replica left by precipitates not extracted is presented in Figure 6. The extracted precipitates were frequently found to be transparent to the electron beam. Electron-diffraction patterns taken from such precipitates permitted two different plates to be identified as  $WO_2$ . The average size of the precipitates was found to be approximately 6 x 1 x 0.1 micron, and they were precipitated in such a way that the plane of the plate was the (001) cleavage plane of the matrix. Similar platelets were extracted from the fracture surface of Specimen 3 broken at room temperature.

#### W-3Re

The effect of 3 at. % rhenium on the failure mode of tungsten was to greatly increase the amount of cleavage, all recrystallized specimens tested above ambient exhibiting approximately 50% cleavage and 50% grain-boundary failure (see Table 1, Specimens 18, 19, and 20). This is somewhat surprising, since the grain size in this material was appreciably smaller than that in the unalloyed tungsten annealed at 3500 F, yet in the tungsten it was found that the larger grains tended to cleave rather than the

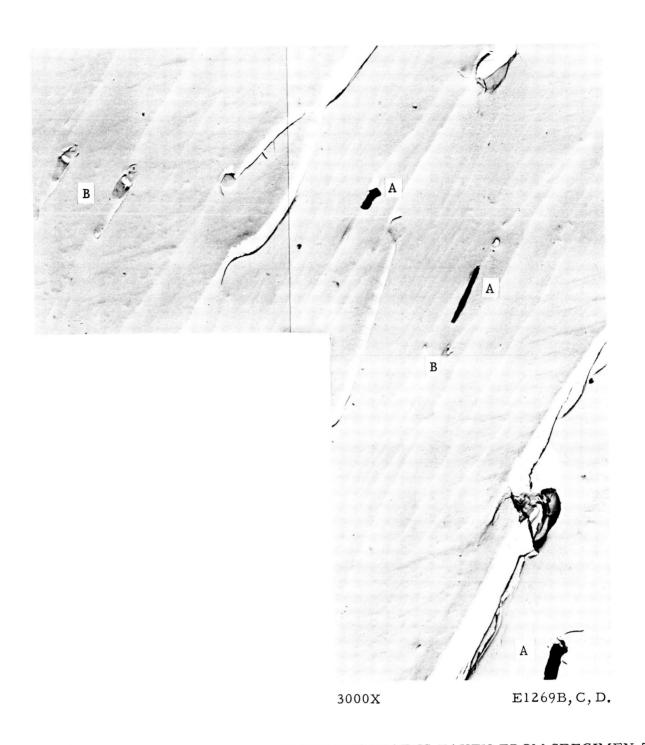


FIGURE 6. MONTAGE OF ELECTRON FRACTOGRAPHS TAKEN FROM SPECIMEN 5

Dark Areas A are extracted plates of WO2; Areas B are impressions on replica left by similar plates.

Unaller. From this evidence alone, it would be predicted that the smaller grained W-3Re would have a greater tendency to part by grain-boundary failure; this was found not to be the case. The issue is confused by the fact that the W-3Re specimens were fractured at 300 to 400 F, whereas the tungsten was tested at ~750 F. However, the specimens broken at room temperature (last three items in Table 1) also tend to a greater amount of cleavage as rhenium is added, although the amount of cleavage is greater over all at the lower temperature. Thus, adding rhenium produces more cleavage failure, and there is some evidence that decreasing the test temperature also leads to a greater percentage of cleavage failure.

Cleavage facets again tended to be in the larger grains and occasionally showed chevron markings. Because of the small grain size, there was no evidence for a change in the nature of the river lines of a particular cleavage facet as it spread from the tension side of the specimen to the compression, but individual cleavage facets showed the same over-all trend. For example while cleavage facets from the tension side of a specimen show normal unidirectional river lines, Figure 7 shows a typical cleavage facet from the compression side of Specimen 18, where well-defined markings are visible perpendicular to the general direction of cleavage.

Under the optical microscope, the precipitate morphology seemed very similar to that in tungsten, but in general the boundaries were much cleaner than those of the unalloyed tungsten and devoid of any continuous interference films. The boundaries contained globular precipitates and occasional isolated examples of platelike precipitates thin enough to produce interference effects. Examples of the shapes of the latter type of precipitates are shown in Figure 8. To determine whether such platelets were present prior to testing or were formed on cooling from the test temperature, the fracture surface of the specimen broken at room temperature was examined. No evidence of any grain-boundary features giving rise to interference effects were apparent under the optical microscope, and no precipitates similar to those in Figure 8 were found on replicas. This strongly suggests that grain-boundary precipitates such as those in Figure 8 are formed after fracture and are not representative of any grain-boundary film existing prior to the test. The fracture surface of the specimen broken at room temperature showed only globular precipitates.

#### W-5Re

The fracture characteristics of W-5Re were the same as for W-3Re apart from the smaller grain size, and possibly an even greater tendency for cleavage as opposed to grain-boundary failure (see Table 1). The exposed grain boundaries had in general more precipitates present than did the W-3Re and about the same as did the unalloyed tungsten. Such precipitates were again globular as illustrated in Figure 9, which is a replica fractograph taken from the room-temperature fracture of Specimen 26. On the surfaces exposed by fracture at higher temperatures however, precipitates similar to those shown in Figure 10 were observed on electron replicas. Such features are again believed to have been formed after fracture.



400X 18608

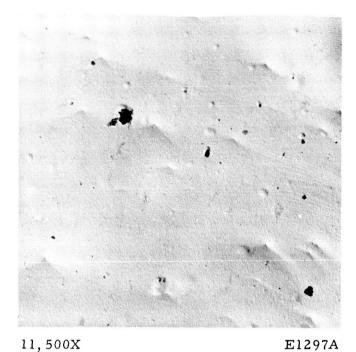
FIGURE 7. CLEAVAGE FACET ON COMPRESSION SIDE OF FRACTURE SURFACE OF SPECIMEN 18 SHOWING WELL-DEFINED MARKINGS PERPENDICULAR TO THE DIRECTION OF CRACK PROPAGATION



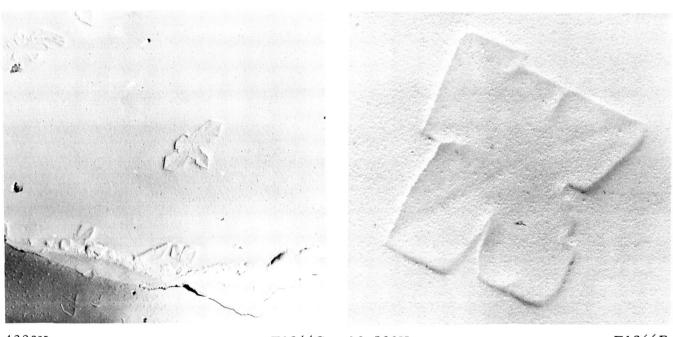
15,500X E1270B

FIGURE 8. EXAMPLES OF GRAIN-BOUNDARY PRECIPITATES IN W-3Re REVEALED BY ELECTRON REPLICA FRACTOGRAPHY

Such precipitates are believed to have been formed after fracture at high temperature.







4300X E1266C 19,500X E1266B

FIGURE 10. ELECTRON REPLICAS OF GRAIN-BOUNDARY PRECIPITATES REVEALED ON SPECIMEN 26 FRACTURED AT HIGH TEMPERATURE

# Wrought Materials, W-3Re and w-5Re

The nonrecrystallized materials were capable of bending at or about room 'emperature, some 300 F lower than the recrystallized material. Under the optical microscope, the fracture surface of wrought materials was found to consist of regions of delamination. Figure 11 shows metallographic sections through the fractures of four specimens showing delamination to different extents as a result of composition and heat-treatment differences. The structures of Specimens 21, 24, 14, and 16 are presented in Figure 12 and are arranged in increasing extent of recovery. It can thus be seen by comparing Figures 11 and 12 that the extent of delamination is clearly associated with the degree of recovery, decreasing in the order 21, 24, 14, and 16. Furthermore, the transition temperature of these specimens is also seen (from Table 1) to increase following this progression. Thus, a greater tendency toward a failure by delamination accompanies an increased capability for bending at lower temperatures. The mechanism behind this effect is made clear by Figure 13, which shows a metallographic section through the thickness of Specimen 16. Transverse cracks propagating in a direction which could ultimately lead to failure have been arrested by the layered structure, the laminations of the incompletely recrystallized structure serving to arrest the propagating crack. The same mechanism has been found to operate in sheet samples of thoriated tungsten-rhenium alloys. (5)

Examination of the fracture surfaces of wrought samples under the optical microscope shows the expected layered structure, with apparently 10 to 30 layers in a specimen thickness. Electron replicas of the fracture surface however show the structure to be layered on a much finer scale. A replica taken from a surface exposed by delamination is shown in Figure 14a and is completely grain-boundary surface. Closer to the actual transverse fracture, however, the laminate itself had a stepped appearance where individual grains had cleaved across their thickness, as shown in Figures 14b and 14c.

#### DISCUSSION

The effect of rhenium on the ductile-brittle transition temperature of recrystallized W is difficult to evaluate because rhenium has a marked effect on grain size which has in turn a marked but unpredictable effect on transition temperature. For example, in steel, refining the grain size depresses the transition temperature, but in medium and coarse grained chromium, increasing the grain size depresses the transition temperature. There are two plausible rationalizations for this difference, one of which is that in steel the tensile transition temperature represents the crack-propagation transition temperature, whereas in chromium the tensile transition represents the crack-initiation transition. It may well be that the two types of controlling processes have reverse grain-size dependencies. On the other hand, it may be that for all materials, the generalized curve of transition temperature versus grain size passes through a maximum value. Such a relationship has been documented by Grant for iron(6) and is reproduced in Figure 15. The behavior of steel and chromium could then be considered as representative of opposite sides of the maximum. However, such detailed studies of transition temperature versus grain size have not been performed for a sufficient number of materials for the question to be resolved. The issue is

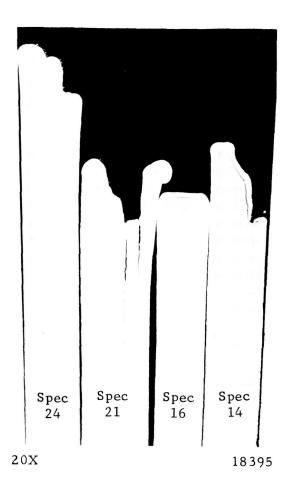
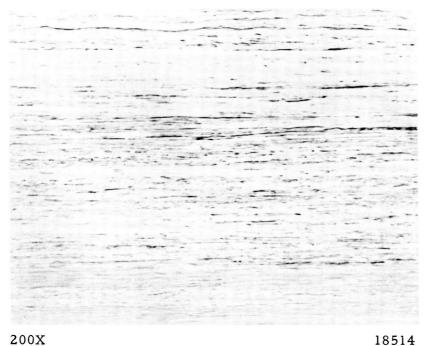


FIGURE 11. FRACTURE PROFILES OF FOUR WROUGHT SPECIMENS SHOWING DELAMINATION TO DIFFERENT EXTENTS

The specimen length is vertical on the page, specimen width is into the page, and specimen thickness is across the page.



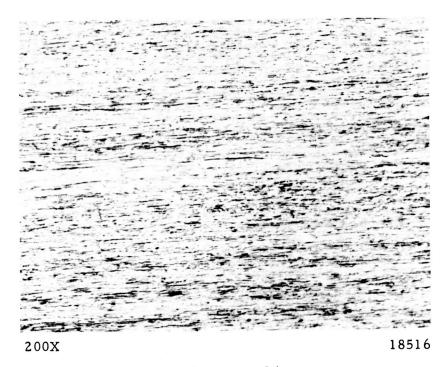
a. Specimen 21



b. Specimen 24

FIGURE 12. METALLOGRAPHIC SECTIONS SHOWING THE EXTENT OF RECOVERY IN THE 4 SPECIMENS OF FIGURE 11

18



c. Specimen 14



d. Specimen 16

FIGURE 12. (CONTINUED)

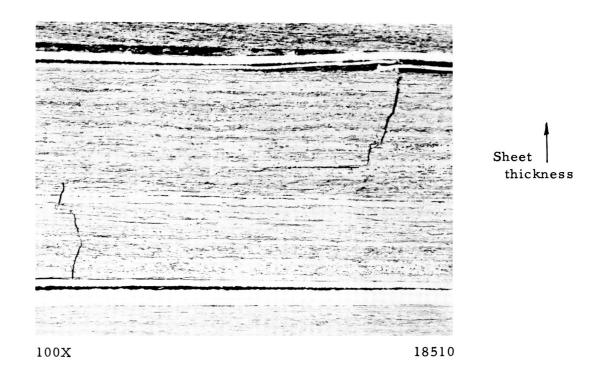
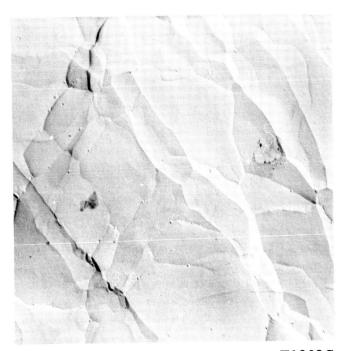


FIGURE 13. METALLOGRAPHIC SECTION THROUGH SPECIMEN 16 SHOWING TRANSVERSE CRACKS ARRESTED AT DELAMINATIONS





11,500X E12

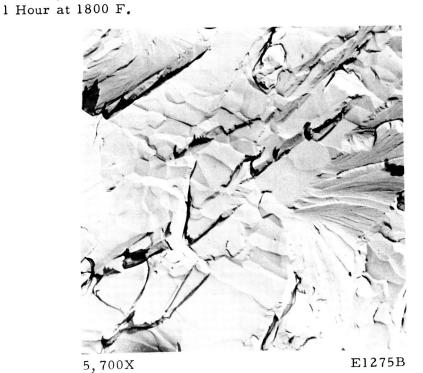
a. Specimen 14, W-3Re Annealed

E1282C

5,700X

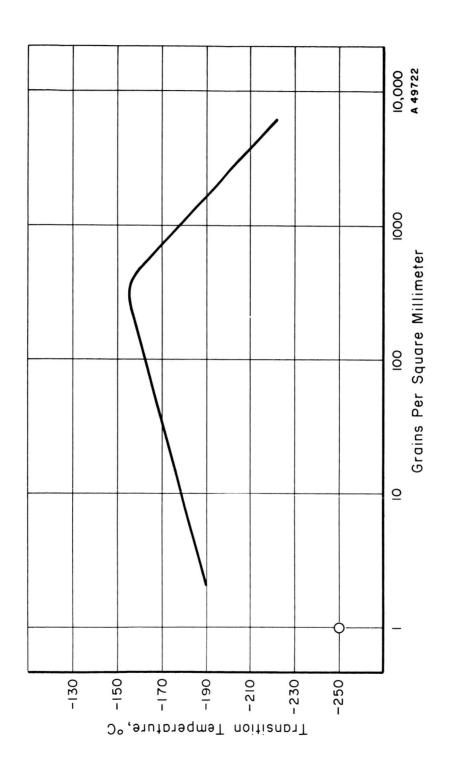
E1276D

b. Specimen 21, W-5Re As Rolled.



c. Specimen 21, W-5Re As Rolled.

FIGURE 14. ELECTRON REPLICA FRACTOGRAPHS TAKEN FROM FRACTURED WROUGHT SPECIMENS



TRANSITION TEMPERATURE VERSUS GRAIN SIZE FOR PURE IRON. REFERENCE (6). FIGURE 15.

particularly relevant for the present results, where increasing the grain size in unalloyed tungsten depresses the transition temperature, behavior typified by the left side of Figure 15, while the finer grained rhenium alloys have a lower transition than the lowest determined for unalloyed tungsten. One explanation of the transition behavior of these materials could be that the coarse-grained tungsten lies to one side of the maximum in the curve of transition temperature versus grain size, while the finer grained W-Re alloys lie to the other side. This explanation lacks complete consistency, however, since the W-5Re alloy has a finer grain size and a slightly higher transition temperature than does the W-3Re alloy, whereas for the above explanation to hold, the transition temperature should have been lower. The discrepancy in this respect, however, is only 50 F (450 F vs. 500 F, see Table 1), which could perhaps have arisen through experimental scatter, although a further perturbing influence is the much cleaner appearance of the exposed grain boundaries in the W-3Re alloy compared with those of either the unalloyed tungsten or the W-5Re. This could be the controlling factor in explaining the lower transition temperature of the W-3Re compared with the W-5Re alloy.

In addition to the effect of rhenium on the recrystallized grain size, the fracture mode was found to be predominately cleavage in the rhenium alloys but grain boundary in unalloyed tungsten. This change in fracture mode could be either a result of the change in grain size or a separate effect in its own right. There is some support for the former possibility insofar as there is a slight tendency for the unalloyed tungsten to show more cleavage at the smallest grain size. Furthermore, alloying seems to produce no major change in the morphology of the grain-boundary precipitates such as would be necessary to explain the readiness of the grain boundaries to part in tungsten but not in the alloys. However, it is not apparent why the tungsten should cleave preferentially in the larger grains, and yet for the much smaller grained alloys to fail almost entirely by cleavage. This phenomenon may be a result of solid-solution strengthening and a finer over-all grain size, which together raise the stress at which fracture in the alloys is initiated to a level at which a running crack is propagated with ease through the matrix. By comparison, in unalloyed tungsten the weaker grain boundaries form a preferential path for fracture propagation.

In view of the evidence for slip-induced cleavage failure, the preference for large grains to cleave may arise, as has been suggested in earlier models of yielding, from the larger stress concentrations which can be obtained due to the slip bands of greater length which exist in the larger grains. The preference for crack initiation at grain corners on triple points must arise from the stress-concentrating effects of such "internal notches".

It thus seems that that through one mechanism or another, grain-size variation plays an important part in determining the fracture mode of tungsten and W-Re alloys. To delineate the true effect of rhenium on the ductile-brittle transition temperature of tungsten, the transition temperature of alloys of the same grain size needs to be determined, and even then an interpretation of the fracture mechanisms may still be confused by the unavoidable solid-solution strengthening which would still cause the fractures to propagate at different stress levels.

Little can be said concerning the change which occurs as river lines cross from the tension side of a specimen to the compression side. The observation is of interest in itself however, and it probably represents a change in nature of cleavage propagation arising as the crack proceeds into a region which has been stressed differently from that at which the crack was initiated. The change may or may not represent a change in the previously active slip system.

One further observation which may be of extreme importance to the propagation of cleavage in tungsten is the WO<sub>2</sub> precipitates shown in Figure 5 which were extracted from a cleavage facet. It is not likely that these platelets were formed during the cooling from temperature, but they were probably there prior to fracture since the river lines in the cleavage facet seem in some instances to be perturbed in the region of the precipitates. Furthermore, similar platelets were observed on room-temperature cleavage facets. Those platelets whose major dimensions lie in the cleavage plane must be regions of high stress concentrations perpendicular to the cleavage plane. Similar platelets have been observed in brittle fractures of chromium and molybdenum. (7) Such regions of built-in stress concentration may play a significant role in the failure of the refractory metals, since the Group VIA metals, which have low solubility for interstitials and are therefore most likely to contain such second-phase particles, are the very ones most susceptible to cleavage failure.

It is clear that in the wrought alloys the lamination of the worked structures largely determines the resistance of such material to cleavage failure. The mechanism by which the delamination occurs is apparently grain-boundary failure during rolling, since the surfaces of the delaminations are found to be entirely grain boundary. Hence, the effect of rhenium on the as-cast grain size may significantly affect the number and width of laminations, as well as the stage of rolling at which delaminations first appear. Therefore, to optimize the beneficial effect of rhenium (which should be particularly useful practically since rhenium retards recovery), future studies should take into consideration such factors as the as-cast grain size and rolling temperatures and reductions with respect to the resulting laminated structure.

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- (7) A. Gilbert, unpublished work.